

IMAGE ANALYSIS FOR THE CHARACTERIZATION OF MATERIALS FOR HIGHWAY CONSTRUCTION

PHASE I PROGRESS REPORT

NOVEMBER 30, 1992

IOWA DOT PROJECT HR-346

**Sponsored by the Highway Division of the
Iowa Department of Transportation and the
Iowa Highway Research Board**

Department of Forestry / Iowa State University

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PHASE 1 PROGRESS REPORT

NOVEMBER 30, 1992

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"The opinions, findings and conclusions expressed in this publication are those of the authors and not necessarily those of the Highway Division of the Iowa Department of Transportation."

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ABSTRACT

The major objective of this project is to evaluate image analysis for characterizing air voids in P.C.C. and A.C. and aggregate gradation in A.C.

Phase 1 of this project has concentrated on evaluation and refinement of sample preparation techniques, evaluation of methods and instruments for conducting image analysis, and finally, analysis and comparison of a select portion of samples.

Preliminary results suggest a strong correlation between the results obtained from the linear traverse method and image analysis methods for determining per cent air voids in concrete.

Preliminary work with asphalt samples has shown that damage caused by the high vacuum of the conventional SEM may be too disruptive. Alternative solutions have been explored, including confocal microscopy and low vacuum electron microscopy. Additionally, a conventional high vacuum SEM operating at marginal operating vacuum may suffice.

INTRODUCTION

The following report summarizes the research activities conducted on Iowa Department of Transportation Project HR-346 for the period December 15, 1991 through December 31, 1992. The objective of this research is to evaluate the application of image analysis for characterizing air voids in P.C.C. and A.C. and aggregate gradation in A.C.

RESEARCH ACTIVITIES

Sample Preparation

The necessary preparation techniques for P.C.C. cores to be analyzed for air void distribution have been refined. While the equipment provided by the Materials Analysis and Research Laboratory at Iowa State University is capable of producing quality surfaces, routine production would require better equipment. Specifically, a large scale automated polisher would greatly facilitate concrete and asphalt analyses.

The first years work further showed that the amount of time necessary for grinding a sample prior to final polish was highly dependant upon the quality of surface provided by the diamond cutoff saw. A slow cut on a quality blade can result in considerable time savings. Further, a slower cut results in extended saw blade life and lower consumption of grinding discs.

A satisfactory procedure for concrete cores is as follows:

- 1) Grind core with 60 grit disc until uniform (2-15 minutes depending on saw cut).
- 2) Grind core with 180 grit disc until uniform (approximately 2 minutes).
- 3) Grind core with 400 grit disc until uniform (approximately 2 minutes).
- 4) Grind core with 600 grit disc until uniform (approximately 2 minutes).
- 5) Polish core with 5 micrometer alumina (approximately 2 minutes).
- 6) Polish core with 1 micrometer alumina (approximately 2 minutes).

It should be noted that a larger scale automated polisher would shorten this procedure as more uniform and higher grinding/polishing loads could be applied. Additionally, 3-4 samples could be simultaneously prepared.

Similarly, A.C. can be ground and polished by the above procedure. Further work to determine if lowering the temperature of the sample would have a beneficial effect is planned.

Imaging Devices

Four types of imaging devices have been assessed to determine the optimal method for obtaining images for analysis. These instruments are:

Conventional Scanning Electron Microscope

A conventional scanning electron microscope (SEM) operated in the backscattered electron mode (BSE) was used for both P.C.C. and A.C. As previously reported, the backscatter imaging mode derives its signal intensity based upon the atomic number of the specimen. By coating the sample with a heavy layer of gold, it was hypothesized that very high contrast between the concrete and the voids could be obtained. Analysis of the images thus obtained could be accomplished by an image analyzer. A variety of accelerating voltages, working distances, beam currents, and magnifications were assessed to determine optimal conditions for conducting these analyses. Figure 1 illustrates the exceptional quality of contrast obtained by imaging cores in this fashion.

Low Vacuum Scanning Electron Microscope

A number of low vacuum SEM instruments were evaluated to determine their suitability. Since these instruments are not available locally, only a limited amount of work could be performed on these systems. The major difference of these instruments over the

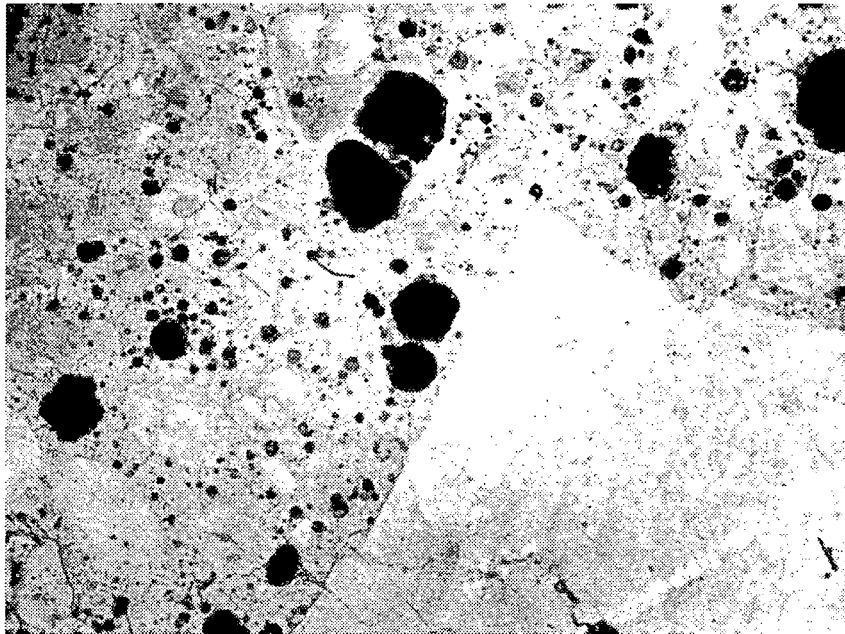


Figure 1. Backscattered electron micrograph of voids in concrete.

conventional SEM is there ability to operate at low vacuum thus eliminating the high vacuum pressure on specimens that can cause micro-cracking in concrete and disruption of the sample surface in asphalt. Figures 2 & 3 depict images of concrete and asphalt (respectively) obtained from a low vacuum scanning electron microscope. These images were not coated with gold to ensure that micro-cracking, if present, could not be attributed to the sputter coating device. Micro-cracking was not apparent, but the numerous issues concerning applicability of the low vacuum SEM still remain to be resolved.

Confocal Light Microscope

A confocal light microscope owned and operated by the University of Iowa Microscopy Facility was also used to examine P.C.C. and A.C. This microscope consists of a laser-beam illumination device attached to a conventional light microscope. The unique feature of a confocal microscope is its ability to image planes of specified thicknesses in a specimen as illustrated in Figure 4.

A confocal micrograph of a concrete core is shown in Figure 5 and micrographs of successive scans through an asphalt specimen are shown in Figures 6 & 7.

CCD/Macro Camera

A charged coupled device camera (CCD) equipped with a macro lens was also evaluated for image formation. This device was considered as it is able to image an entire core at one time. The resolution of the image is too low for adequately characterizing entrained air but more than adequate for entrapped air voids.

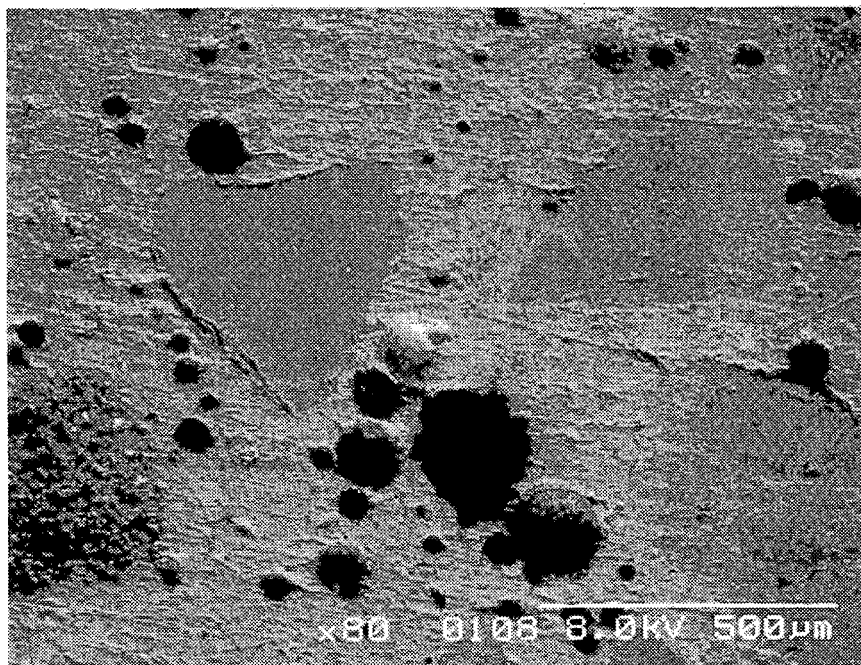


Figure 2. Low vacuum SEM micrograph of concrete.

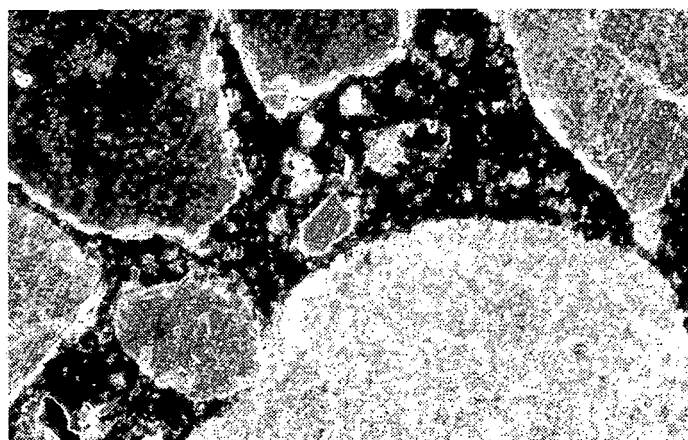


Figure 3. Low vacuum SEM micrograph of asphalt.

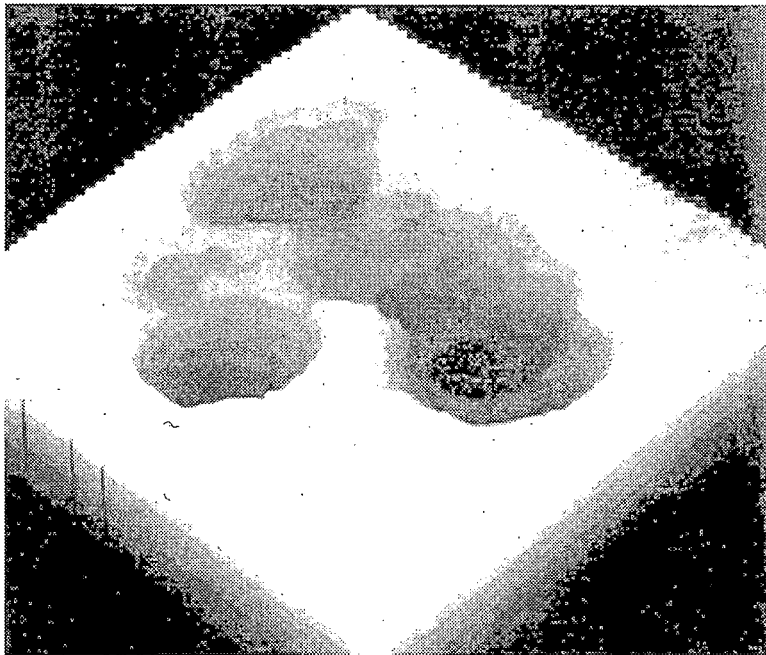


Figure 4. Confocal image depicting several planes of varying depths. Image reprinted from Confocal Microscopy. T.Wilson, 1990.

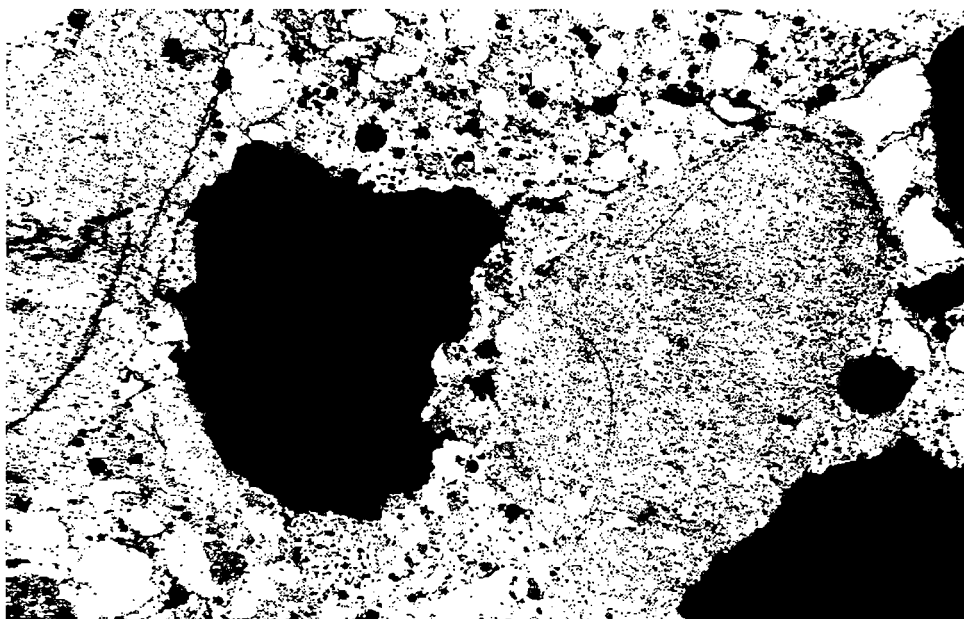


Figure 5. Confocal micrograph of concrete core showing air voids.

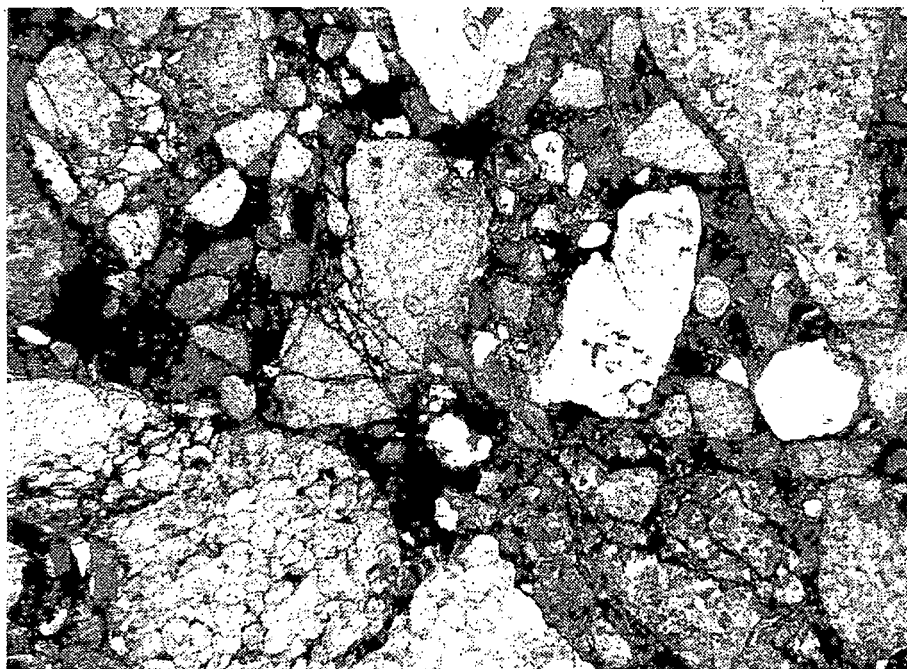


Figure 6. Confocal image of asphalt showing aggregate and voids.

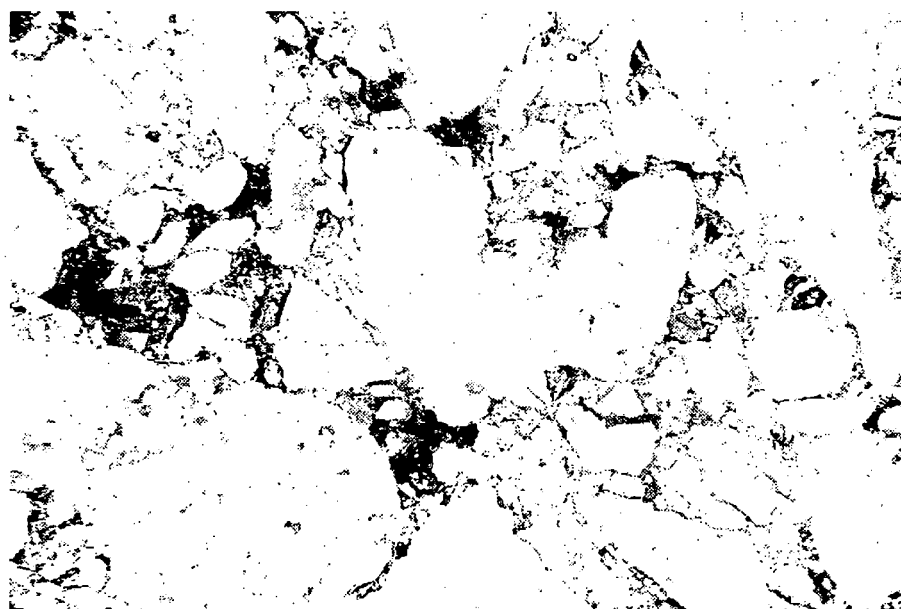


Figure 7. Confocal image of successive scan showing void structures.

Image Analysis Systems

To date, twelve image analysis systems have been evaluated for applicability to our needs. The following is a list of those systems:

- 1) LECO analyzer
- 2) Lemont Scientific D10
- 3) IBAS (Zeiss)
- 4) OASYS
- 5) PV-Wave (Project Vincent)
- 6) Khoros (Project Vincent)
- 7) Jandell (JAVA)
- 8) Compix Imaging
- 9) Fision (formerly Kevex)
- 10) Universal Imaging
- 11) Image/NIST
- 12) Nikon

Comparative Analyses

Eight cores of P.C.C. obtained from the IDOT have been analyzed for air void content by image analysis. In addition, seventeen cores obtained from a SHRP program are currently being evaluated by image analysis.

Preliminary work on A.C. has been completed and analysis of specimens will be conducted upon receipt of the samples from the IDOT.

RESULTS AND DISCUSSION

Sample Preparation

Techniques for preparing samples have been refined such that adequate samples can be prepared given the available equipment. It is obvious that faster and better methods can be developed but would require investment in better equipment.

Imaging Devices

The SEM, ESEM, confocal light microscope, and CCD/Macro, are suited to perform the desired work. There are advantages and disadvantages associated with each and further work would more clearly define the best course of action. The following is a brief summary of each instrument.

Conventional Scanning Electron Microscope

The majority of work during Phase I was performed on the conventional SEM and it has proven sufficient for this work. The original plan to scan micrographs via an ancillary vidicon camera was replaced by a direct digitization method thus yielding superior images for subsequent analyses. Program routines were then written to streamline data analysis, convert image formats, and provide long-term archival data storage via the mainframe computer at Iowa State Universities Computations Center. These images, obtained from the microscope in digital format, can now be translated into the proper format suitable for analyses by any image analysis system. Further streamlining and gains in efficiency are planned for Phase II work. Specifically, a more direct link of the OASYS image analyzer to the SEM would greatly improve digitization, speed, and analyses.

Low Vacuum Scanning Electron Microscope

The low vacuum SEM has some inherent advantages that should be recognized if large-scale routine analyses are envisioned. Phase I evaluation of various low vacuum systems has proven that "wet" samples can be easily handled by these microscopes so that analyses could be performed during the construction of a road. Furthermore, even with cores that have been completely cured, the need for pre-pumping of the core prior to analyses is eliminated.

Confocal Microscope

The confocal microscope, with its ability to achieve magnifications as low as 1X, combined with its ability to scan prescribed planes of depth, offers some unique capabilities for characterizing concrete and asphalt. Additionally, because this is a light microscope based system (not enclosed in a vacuum chamber), one can freely move about on the sample surface to any desired location.

Charged Coupled Device Camera

Originally, evaluation of the CCD camera was not planned as a part of this research but the occasional presence of large entrapped air voids merits pursuing its capabilities. As this camera allows one to image the entire core simultaneously, classification of the amount of entrapped air can be determined in a matter of seconds.

Comparisons between Linear Traverse and Image Analysis

The majority of work performed during Phase I was conducted using the conventional SEM. This system is proving to be sufficient to analyze air void distribution in concrete. Figure 8 shows the results from a series of cores analyzed by linear traverse and by SEM/Image Analysis. These preliminary results are very encouraging and indicate there may be a strong correlation between linear traverse and SEM/Image Analysis. The cores for Figure 8 were from IDOT samples and the linear traverse results were performed by an outside testing laboratory and IDOT personnel.

Further work is planned for comparative analysis using cores obtained from a SHRP project (linear traverse performed by Michigan State University). To date, only one of these comparative analyses has been performed due to discrepancies found in the linear traverse data. The comparative results between linear traverse and image analysis for

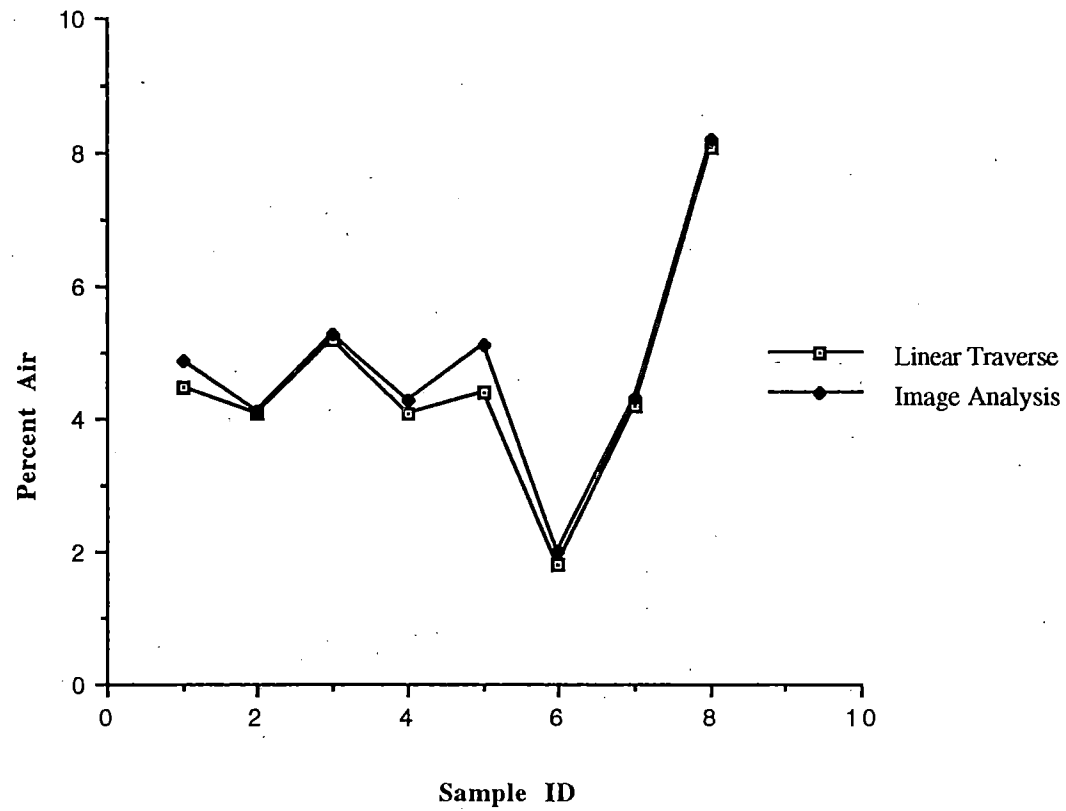


Figure 8. Comparison of linear traverse and image analysis results of percent air void.

this core (Core E9A) are shown in Figure 9 and point out key differences between the two analyses. Figure 9 shows the distribution of voids by size and indicates a large difference between the two methods. As shown, the majority of voids measured by linear traverse are greater than 250 micrometers in diameter while a majority of voids measured by image analysis are less than 250 micrometers.

To investigate further, we can consider the typical air entrainment of a core of concrete containing a similar water/cement ratio. Table 1 shows the expected distribution for a number of ratios including .55. In general, this table indicates that approximately 85% of the air voids should be less than or equal to 100 micrometers in diameter. Further examination of Figure 9 shows that approximately 82% of the air voids measure by image analysis are less than or equal to 100 micrometers. In comparison, the linear traverse results indicate that only 20% of the air voids measure less than or equal to 100 micrometers.

Further, Figure 10 summarizes the void distribution over the entire bank of 17 samples from the Michigan data and shows that only 11% of the void total consists of voids in the category of 100 micrometers or less.

Additionally, the data from the Michigan study contains a number of numerical discrepancies that are currently being reviewed by the Michigan investigators, the results of which should be forthcoming.

FURTHER DISCUSSION

The work performed during Phase I of this research assesses the capabilities of a number of instruments and the potential for these instruments to perform the desired tasks. The key issue to consider is whether or not these systems are capable of providing images sufficient for performing image analyses. The work to date indicates that these systems are capable of providing such images. Further work is needed to better define the amount of sampling required and the most efficient manner for performing the analyses.

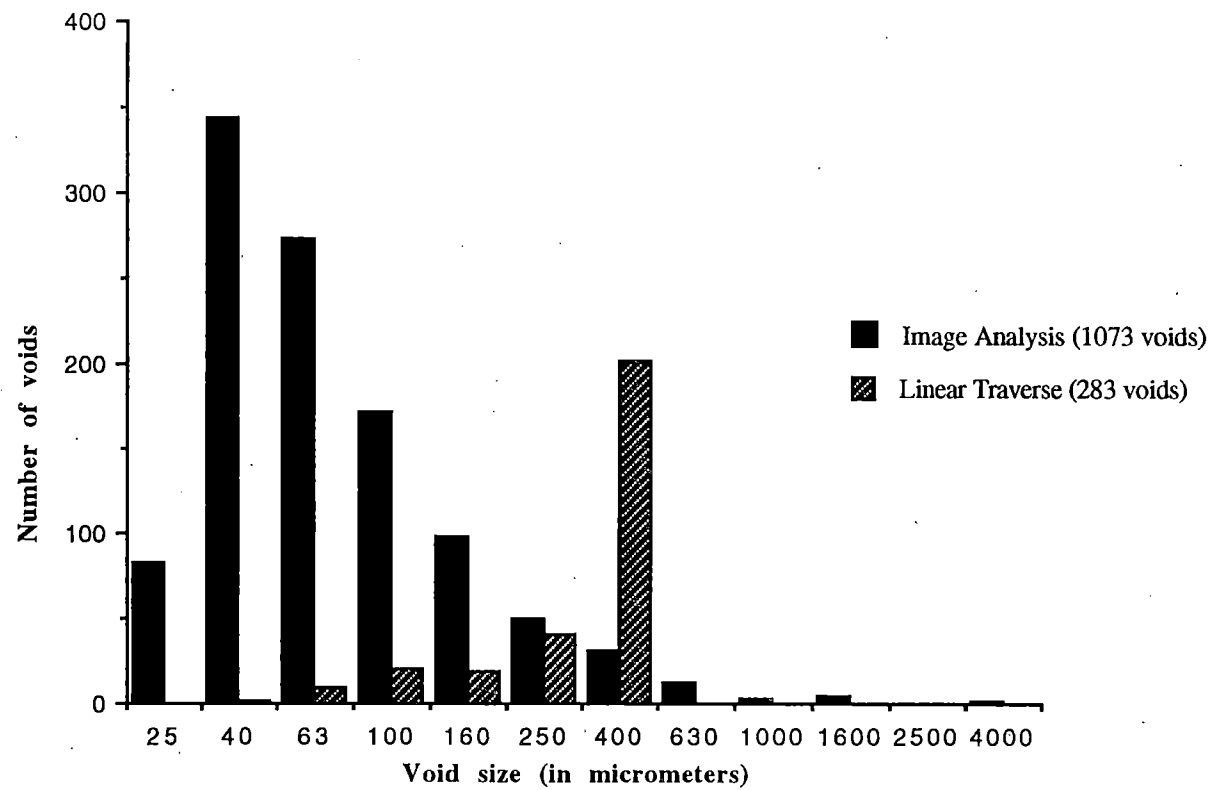


Figure 9. Comparative void analysis for core E9A.

Table 1. Typical air void distribution of entrained air concretes.

Diameter of Bubbles in micrometers	Number of Bubbles per cc of Concrete		
	w/c=0.35	w/c=0.55	w/c=0.75
20	255,234	73,240	4000
40	128,386	39,685	20,661
60	26,035	16,285	7878
80	13,183	8191	3234
100	7348	5215	1589
120	1847	4002	1103
140	1354	889	694
160	812	832	531
180	639	523	779
200	1271	1381	1672
250	118	129	196
300	33.7	120	136
350	15.3	61.1	118
400	59.8	94.2	57.9
450	47.7	54.1	31.8
500	68.4	104.0	79.5
600	4.3	14.9	18.2
700	3.1	17.8	17.8
800	20.3	1.6	1.7
900	---	1.3	1.3
1000	---	9.7	8.7
2000	---	1.0	2.0
Total	436,480	150,852	42,811
Air content (%)	4.8	5.2	5.3
Paste content (%)	41.2	26.9	24.1
Number of bubbles per cc of paste	106.0 E+04	56.0 E+04	1.8 E+04

a) Data are from J. E. Backstrom, et al., "Origin, Evolution, and Effects of the Air Void System in Concrete, Part III Influence of Water-Cement Ratio and Compaction, " *Proceedings ACI*, 55, 359-375, 1958.

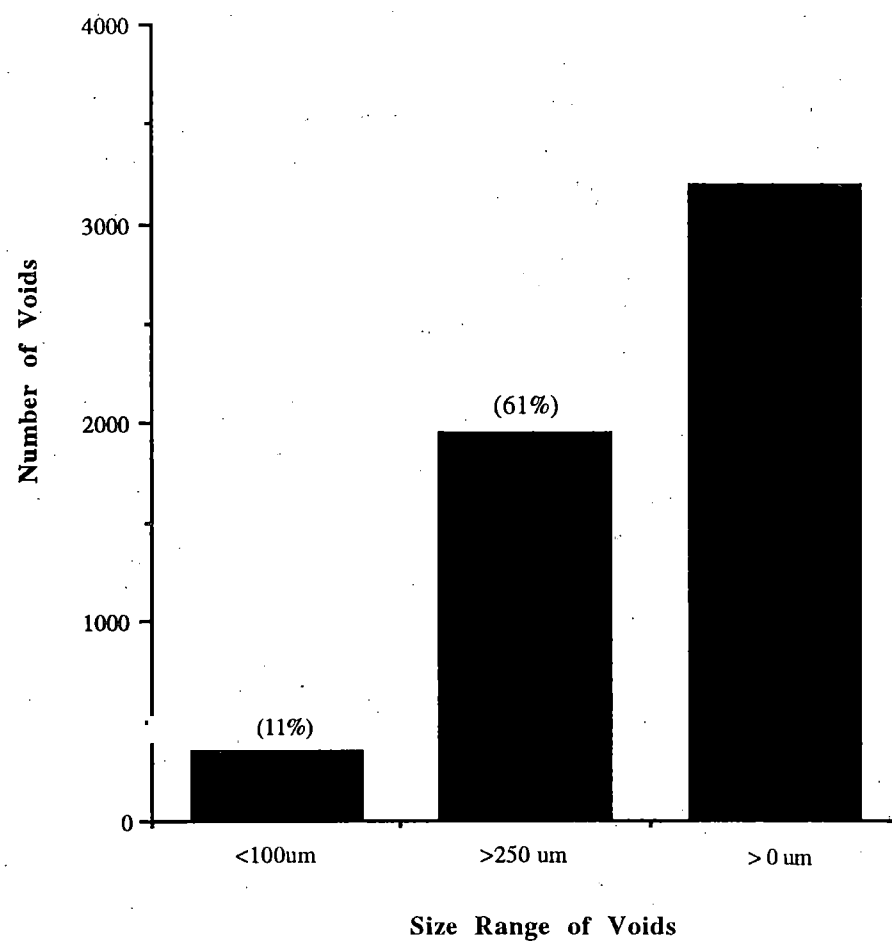


Figure 10. Composite void distribution of Michigan data from linear traverse.

One issue of void analyses meriting further discussion is void size and distribution. The Michigan State University data presented results from linear traverse for not only the total air void content but also the void count in the various size ranges. This information provides a greater knowledge base about the sample and could possibly yield important information about service life based upon the size and distribution of air bubbles. Appendix 1 contains an example of an IDOT core analyzed and sorted into various specified size ranges by image analysis.

Additionally, this particular core displayed a wide range of entrapped air when sectioned at quarter inch increments through the core. The entrapped air content measured 1.16, 3.94, 8.19, and 3.25 % respectively on these quarter inch increments. Using the traditional approach by linear traverse, the void sizes would not be separated into various classes and the influence of large amounts of entrapped air would not be so apparent. Further, the use of the CCD camera allowed us to determine these entrapped air void amounts in minutes for the entire set of 4 cores.

By pooling the data from the analyses by SEM and the CCD camera images, a total air content value for the top surface was determined to be 4.34% by image analysis and 4.22% by linear traverse. Linear traverse values were not measured on the subsequent increments.

SUMMARY

Comparisons between linear traverse and image analysis have shown promise and further comparisons are warranted. Some difficulties have been encountered with respect to the reliability of the linear traverse information obtained from Michigan State University and these difficulties will need to be resolved before work progresses much further with these cores.

Further comparisons will provide enough data to perform statistical analyses and determine correlation coefficients for image analysis. Efforts to determine spacing factor measurements and size distribution histograms of air void analyses also need further study.

More time and emphasis will be focussed on asphalt analyses during Phase II work. Continued work on imaging and analysis of asphalt on the conventional SEM is needed although the use of a low vacuum SEM or a confocal microscope may prove to be better alternatives due to the disruption of the samples caused by the high vacuum system. Comparative analyses between these various systems would be beneficial.

Lastly, further improvements in existing instrumentation to enhance the efficiency and speed of analyses will continue under Phase II work.

Other Benefits

The superb resolution, combined with the enhanced depth of field and relatively large surface area scanned by the SEM has yielded additional information that may prove more valuable than reporting of the air content alone.

During Phase I work, a variety of atypical phenomena were revealed as cores were analyzed. On occasion, certain cores would exhibit such things as:

- 1) Stratification of air bubbles.
- 2) Highly variable air content with a core.
- 3) "Pop-out" caused by reactive flyash.
- 4) Cores with uneven distribution of large aggregate.
- 5) Cores with uneven distribution of small aggregate.
- 6) Pores filled with gel.
- 7) Pores filled with ettringite.

The diagnostic value and interpretation of this information undoubtedly would require further investigation and study but could be of major benefit. The process of gathering this information is not difficult, nor is it time-consuming. Presumably,

idiosyncracies found in asphalt samples are expected and in fact, preliminary examinations of asphalt have shown areas with variable aggregate distributions.

ACKNOWLEDGMENTS

We would like to extend our thanks to the people who have contributed to this work. A special thank you is extended to the IDOT personnel for their input, suggestions, and generous help in conducting this work.

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APPENDIX 1

Image analysis results by scanning electron microscope and
CCD camera imaging of concrete core CP1

TOTAL SAMPLE

1322 FEATURES AFTER FRAME 10 AREA ANALYZED = 1.87E+00 SQCM.

FEATURES PER FRAME: MEAN = 132. SIGMA= 38. [STANDARD]

%FEAT. AREA PER FRAME MEAN = 3.18E+00 SIGMA=1.14E+00 [DEVIATIONS]

EXTREMES IN FEATURE LARGEST SMALLEST

PHYSICAL MEASUREMENTS: -----

PARTICLE AREA (SQ.UM.) 3.16E+05 1.53E+02

WIDTH TO LENGTH RATIO 8.74E-01 2.91E-02

WIDTH (UM) 2.83E+02 7.81E+00

LENGTH (UM) 2.58E+03 1.95E+01

ORIENTATION (DEGREES) 1.75E+02 0.00E-01

PERIMETER (UM.) 5.34E+03 4.10E+01

LINE SCAN IMAGE ANALYSIS AREA AND PERIMETER SUMMARY:

FOR ALL PARTICLES

	TOTAL		TOTAL		TOTAL		
LVL	AREA	AREA	E.PERIM	E.PERIM	I.PERIM	I.PERIM	I/E PERIM
	(SQ.UM.)	FRAC	(UM.)	FRAC	(UM.)	FRAC	RATIO
0	2.40E+04	0.004	2.83E+02	1.000	3.22E+03	0.500	0.011
1	5.93E+06	0.996	2.83E+05	1.000	3.22E+03	0.500	0.011

WIDTH (UM) DISTRIBUTION FOR ALL TYPES MACR = 0

AVG=3.79E+01 DEV=2.65E+01 ZDEV=6.99E+01 MEDIAN=2.77E+01 MOST PROB.=2.05E+01

Class	Count	%	0	1	2	3	4
limit	Count	%	0	0	0	0	0
6.3000	0.	0.00E					
10.000	23.	1.74E**					
16.000	156.	11.80E*****					
25.000	420.	31.77E*****					
40.000	340.	25.72E*****					
63.000	247.	18.68E*****					
100.00	93.	7.03E*****					
160.00	35.	2.65E***					
250.00	7.	0.53E*					
400.00	1.	0.08E					

LENGTH (UM) DISTRIBUTION FOR ALL TYPES MACR = 0

AVG=8.44E+01 DEV=1.38E+02 ZDEV=1.63E+02 MEDIAN=5.53E+01 MOST PROB.=5.15E+01

Class	Count	%	0	1	2	3	4
limit	Count	%	0	0	0	0	0
16.000	0.	0.00E					
25.000	15.	1.43E*					
40.000	385.	29.12E*****					
63.000	393.	29.73E*****					
100.00	305.	23.07E*****					
160.00	123.	9.30E*****					
250.00	47.	3.56E*****					
400.00	32.	2.42E***					
630.00	11.	0.83E*					
1000.0	6.	0.45E*					
1600.0	2.	0.15E					
2500.0	2.	0.15E					
4000.0	1.	0.08E					

SAMPLE ID: CORE 41 CP

21-OCT-92

TOTAL SAMPLE

200 FEATURES AFTER FRAME 1 AREA ANALYZED = 7.50E+01 SQCM.
 FEATURES PER FRAME: MEAN = 200. SIGMA=***** [STANDARD]
 %FEAT. AREA PER FRAME MEAN = 1.16E+00 SIGMA=***** [DEVIATIONS]

EXTREMES IN FEATURE LARGEST SMALLEST
 PHYSICAL MEASUREMENTS: -----

PARTICLE AREA (SQ.UM.) 9.74E+06 1.23E+05
 WIDTH TO LENGTH RATIO 7.64E-01 1.62E-01
 WIDTH (UM) 2.74E+03 1.56E+02
 LENGTH (UM) 5.96E+03 4.31E+02
 ORIENTATION (DEGREES) 1.72E+02 0.00E-01
 PERIMETER (UM.) 1.20E+04 1.13E+03

LINE SCAN IMAGE ANALYSIS AREA AND PERIMETER SUMMARY:

FOR ALL PARTICLES

	TOTAL		TOTAL		TOTAL		
LVL	AREA	AREA	E.PERIM	E.PERIM	I.PERIM	I.PERIM	I/E PERIM
	(SQ.UM.)	FRAC	(UM.)	FRAC	(UM.)	FRAC	RATIO
1	8.71E+07	1.000	4.23E+05	1.000	0.00E-01	0.000	0.000

PARTICLE AREA (SQ.UM.) DISTRIBUTION FOR ALL TYPES MACR = 0

AVG=4.36E+05 DEV=9.42E+05 ZDEV=2.16E+02 MEDIAN=2.08E+05 MOST PROB.=1.60E+05

Class	Count	%	0	1	2	3	4	5	6
limit	Count	%	0	0	0	0	0	0	0
1.0E+05	0.	0.00							
2.2E+05	111.	55.50	*****						
4.6E+05	43.	21.50	*****						
1.0E+06	30.	15.00	*****						
2.2E+06	11.	5.50	*****						
4.6E+06	3.	1.50	*						
1.0E+07	2.	1.00	*						

WIDTH TO LENGTH RATIO DISTRIBUTION FOR ALL TYPES MACR = 0

AVG=4.07E-01 DEV=1.31E-01 ZDEV=3.21E+01 MEDIAN=3.82E-01 MOST PROB.=3.75E-01

Class	Count	%	0	1	2	3	4	5	6
limit	Count	%	0	0	0	0	0	0	0
0.1500	0.	0.00							
0.2000	4.	2.00	**						
0.2500	4.	2.00	**						
0.3000	34.	17.00	*****						
0.3500	5.	2.50	**						
0.4000	84.	42.00	*****						
0.4500	14.	7.00	*****						
0.5000	11.	5.50	*****						
0.5500	11.	5.50	*****						
0.6000	5.	2.50	**						
0.6500	19.	9.50	*****						
0.7000	1.	0.50							
0.7500	6.	3.00	**						
0.8000	2.	1.00	*						

WIDTH (UM) DISTRIBUTION FOR ALL TYPES MACR = 0

AVG=3.43E+02 DEV=3.05E+02 ZDEV=8.89E+01 MEDIAN=1.55E+02 MOST PROB.=1.30E+02

Class	Count	%	0	1	2	3	4	5	6
limit	Count	%	0	0	0	0	0	0	0
100.00	0.	0.00							
160.00	110.	55.00	*****						
250.00	0.	0.00							
400.00	21.	10.50	*****						
630.00	48.	24.00	*****						
1000.0	17.	8.50	*****						
1600.0	2.	1.00	*						
2500.0	1.	0.50							
4000.0	1.	0.50							

SAMPLE ID: CORE #1 CP

31-OCT-92

LENGTH (UM) DISTRIBUTION FOR ALL TYPES MACR = 0

AUG=8.30E+02 DEV=7.08E+02 XDEV=8.53E+01 MEDIAN=6.09E+02 MOST PROB.=5.15E+02

Class	0	1	2	3	4	5	6
limit	Count	%	0	0	0	0	0
400.00	0.	0.00					
630.00	110.	55.00	[*****]				
1000.0	43.	21.50	[*****]				
1600.0	33.	16.50	[*****]				
2500.0	9.	4.00	[****]				
4000.0	3.	1.50	[*]				
6300.0	3.	1.50	[*]				